IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

priority Application Serial No. 09/098,760
priority Filing Date ..... June 17, 1998
Inventor..... R.P. Shah
Assignee.....Johnson Matthey Electronics, Inc.
priority Group Art Unit 1753
priority Examiner ..... S. Versteeg
Attorney's Docket No. 32120-div
Title: Methods of Forming Metal Articles (As amended)

PRELIMINARY AMENDMENT

To: Assistant Commissioner for Patents
Washington, D.C. 20231

From: David G. Latwesen, Ph.D. (Tel. 509-624-4276; Fax 509-838-3424)
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AMENDMENTS

In the Specification

Please replace the title with --Methods of Forming Metal Articles--.

At p. 1, before the paragraph at line 6, insert,

--RELATED PATENT DATA

This patent resulted from a divisional application of U.S. Patent Application Serial No. 09/098,760, which was filed on June 17, 1998.--

Please replace the paragraph beginning at line 16 of page 1 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--In accordance with the invention there is provided a high purity tantalum article, such as a sputtering target having substantially uniform texture. In particular, the invention comprises a tantalum sputtering target of at least about 99.95% tantalum and a substantially uniform {100} crystallographic orientation.--.

Please replace the paragraph beginning at line 21 of page 1 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--A process to provide the tantalum sputtering target is disclosed in Application No. 09/098,761 filed on June 17, 1998, the disclosure of said application is expressly incorporated herein by reference. The process comprises--

Please replace the paragraph beginning at line 16 of page 2 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--The rolling reduction per pass is desirably in accordance with a relationship of the minimum reduction per pass, the roll diameter and the desired billet thickness after forging. Generally, the reduction per pass during rolling is about 10% to 20% per pass.--.

Please replace the paragraph beginning at line 24 of page 5 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Strain in equation (2) is high enough to optimize static recrystallization only for thin targets. But even for these targets non-uniformity in strain distribution through a billet

volume may significantly reduce the amount of strain in some areas. Also, demands on capacity of a forging press or rolling mill necessary to provide strains of equation (2) above for large target billets may be too high for some applications. Therefore, there may be restrictions on attainable strains by rolling or forging operations.--

Please replace the paragraph beginning at line 3 of page 11 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Metallic elements by ICP (Inductively Coupled Plasma) or GDMS (Glow Discharge Mass Spectroscopy) analysis.--

Please replace the paragraph beginning at line 5 of page 11 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Billets were upset-forged at room temperature to a thickness of 75 mm. Teflon films of 150 x 150 mm2 and thickness of 1.2 mm were used as lubricants for frictionless upsetting (alternatively frictionless upset-forging can also be performed at 300 deg. C). Thereafter cold rolling with a roll diameter of 915 mm was performed in sixteen passes with partial reductions of 12% per pass along four directions under an angle of 45°.--

Please replace the paragraph beginning at line 14 of page 11 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Coupons across the thickness of the rolled billet were cut from central, mid-radius and external areas and annealed at different temperatures during 1 hour (h) and investigated for structure and texture and photomicrographs thereof are shown in FIGS.

1-6. FIGS. 1-3 are photomicrographs of the center, mid-radial and edge, respectively, showing the fine grain structure of a tantalum target. FIGS. 4-6 are graphs showing {100} crystallographic orientation at the center, mid-radial and edge.--.

Please replace the paragraph beginning at line 9 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 1: Anneal the billet in vacuum.--

Please replace the paragraph beginning at line 11 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 2: Upset-forge billet using teflon as a solid lubricant at room temperature or at 527F to specific height required for rolling.--

Please replace the paragraph beginning at line 11 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 3: Fly-cut surfaces of the forged billet.--

Please replace the paragraph beginning at line 16 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 5: Anneal in vacuum to obtain a fine grain size and uniform texture.--

Please replace the paragraph beginning at line 20 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 1: Upset-forge using teflon to a height such that Mo = 1.0.--

Please replace the paragraph beginning at line 23 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 3: Upset-forge billet using teflon to a final height as required for rolling operation.--

Please replace the paragraph beginning at line 25 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 4: Fly-cut the surfaces of the forged billet.--

Please replace the paragraph beginning at line 32 of page 12 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 1: Anneal the billet in vacuum.--

Please replace the paragraph beginning at line 3 of page 13 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 3: Fly-cut surfaces of the forged billet.--

Please replace the paragraph beginning at line 6 of page 13 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 5: in vacuum to obtain a fine grain size and uniform texture.--

Please replace the paragraph beginning at line 10 of page 13 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 1: Anneal the billet in vacuum.--

Please replace the paragraph beginning at line 12 of page 13 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 2: Upset-forge billet using teflon as a solid lubricant at room temperature or at 572F.--

Please replace the paragraph beginning at line 13 of page 13 with the following clean replacement paragraph in accordance with 37 C.F.R. § 1.121(b)(1)(ii):

--Step 3: Fly-cut surfaces of the forged billet.--

<u>In the Claims</u>

Please replace the indicated claims with the following clean versions of the claims, in accordance with 37 C.F.R. § 1.121(c)(1)(i). Cancel all previous versions of any pending claim.

A marked up version showing amendments to any claims being changed is provided in one or more accompanying pages separate from this amendment in accordance with 37 C.F.R. § 1.121(c)(1)(ii). Any claim not accompanied by a marked up version has not been changed relative to the immediate prior version, except that marked up versions are not being supplied for any added claim or canceled claim.

CLAIMS

Cancel claims 1-4.

5. (New) A method of forming a metal article, comprising:

subjecting a metallic material to upset forging to form the metallic article, the upset forging being conducted at a temperature below a minimum temperature of static recrystallization of the composition of the billet, the upset forging comprising utilization of a lubricant to entirely separate the billet from a forging tool during the forging.

6. (New) The method of claim 5 wherein the lubricant is selected from the group consisting of polytetrafluoroethylene and polyurethane.

7. (New) The method of claim 5 further comprising forming the metal article into a sputtering target.

8. (New) The method of claim 7 wherein the forming the metal article into a sputtering target occurs during the upset forging.

9. (New) The method of claim 7 wherein the forming the metal article into a sputtering target comprises further processing of the metal article after the upset forging.

10. (New) The method of claim 9 wherein the additional processing comprises recrystallization annealing.

11. (New) The method of claim 5 wherein the metal article comprises one or more of Al, Ti, Ta, Cu, Nb, Ni, Mo, Au, Ag, Re, and Pt.

12. (New) The method of claim 5 wherein the metal article comprises Al.

13. (New) The method of claim 5 wherein the metal article comprises Ti.

14. (New) The method of claim 5 wherein the metal article comprises Ta.

15. (New) The method of claim 5 wherein the metal article comprises Cu

16. (New) The method of claim 5 wherein the metal article comprises Nb.

17. (New) The method of claim 5 wherein the metal article comprises Ni.

18. (New) The method of claim 5 wherein the metal article comprises Mo.

19. (New) The method of claim 5 wherein the metal article comprises Au.

20. (New) The method of claim 5 wherein the metal article comprises Ag.

21. (New) The method of claim 5 wherein the metal article comprises Re.

22. (New) The method of claim 5 wherein the metal article comprises Pt.

23. (New) A method of forming a metal article comprising at least 99.95 wt.% tantalum and having a surface, comprising:

forming a billet comprising a composition with includes 99.95 wt.% tantalum utilizing upset forging, the upset forging being conducted at a temperature below a minimum temperature of static recrystallization of the composition of the billet, the upset forging comprising utilization of a lubricant to entirely separate the billet from a forging tool during the forging; and

after the upset forging, the billet being a metal article comprising 99.95 wt.% tantalum and having a surface; the surface having a substantially uniform {100} crystallographic texture and an average grain size of less than 50 microns.

24. (New) The method of claim 23 wherein the lubricant is selected from the group consisting of polytetrafluoroethylene and polyurethane.

25. (New) The method of claim 23 further comprising forming the metal article into a sputtering target.

26. (New) The method of claim 25 wherein the forming the metal article into a sputtering target occurs during the upset forging.

27. (New) The method of claim 25 wherein the forming the metal article into a sputtering target comprises further processing of the metal article after the upset forging.

28. (New) The method of claim 27 wherein the additional processing comprises recrystallization annealing.

29. (New) The method of claim 23 wherein the metal article surface comprises a maximum grain size of less than 50 microns.

30. (New) The method of claim 23 wherein the metal article comprises a thickness, and an average grain size throughout the thickness of less than 50 microns.

31. (New) The method of claim 23 wherein the metal article comprises a thickness, and an average grain size throughout the thickness of about 25 microns.

REMARKS

Claims 1-4 are cancelled, and new claims 5-31 are added. Applicant requests examination of claims 5-31.

Respectfully submitted,

Dated: 12/11/01

By: _____

David G. Latwesen, Ph.D.
Reg. No. 38,533

priority Application Serial No. 09/098,760
priority Filing Date ..... June 17, 1998
Inventor..... R.P. Shah
Assignee..... Johnson Matthey Electronics, Inc.
priority Group Art Unit 1753
priority Examiner ..... S. Versteeg
Attorney's Docket No. 32120
Title: Methods of Forming Metal Articles (As amended)

VERSION WITH MARKINGS TO SHOW CHANGES MADE ACCOMPANYING PRELIMINARY AMENDMENT

In the Specification

The replacement specification paragraphs incorporate the following amendments. Underlines indicate insertions and ~~strikeouts~~ indicate deletions.

The title has been amended as follows:

~~Metal Article With Fine Uniform Structures And Textures And Process Of Making Same~~

Methods of Forming Metal Articles

At p. 1, before the paragraph beginning on line 6, the following is inserted:

RELATED PATENT DATA

This patent resulted from a divisional application of U.S. Patent Application Serial No. 09/098,760, which was filed on June 17, 1998.

The paragraph beginning at line 16 on page 1 has been amended as follows:

In accordance with the invention there is provided a high purity tantalum article, such as a sputtering target having substantially uniform texture. In particular, the invention

comprises a tantalum sputtering target of at least about 99.95% tantalum and a substantially uniform ~~(100) cubic texture~~ {100 crystallographic orientation}.

The paragraph beginning at line 21 of page 1 has been amended as follows:

A process to provide the tantalum sputtering target is disclosed in Application No. _____ ~~filed on even date herewith~~ 09/098,761 filed on June 17, 1998, the disclosure of said application is expressly incorporated herein by reference. The process comprises:

The paragraph beginning at line 14 of page 2 has been amended as follows:

The rolling reduction per pass is desirably in accordance with a relationship of the minimum reduction per pass, the roll diameter and the ~~desire billet~~ desired billet thickness after forging. Generally, the reduction per pass during rolling is about 10% to 20% per pass.

The paragraph beginning at line 25 of page 5 has been amended as follows:

Strain in equation (2) is high enough to optimize static recrystallization only for thin targets. But even for these targets non-uniformity in strain distribution through a billet volume may significantly reduce the amount of strain in some areas. Also, demands on capacity of a forging press or rolling mill necessary to provide strains of equation (2) above for large target billets may be too high for some applications. Therefore, there may be restrictions on attainable strains by rolling or forming operations.

The paragraph beginning at line 3 of page 11 has been amended as follows:

Metallic elements by ICP (inductively Coupled Plasma) or GDMS (Glow Discharge Mass Spectroscopy) analysis.

The paragraph beginning at line 5 of page 11 has been amended as follows:

~~Or GDMS (Glow Discharge Mass Spectroscopy) analysis.~~ Billets were upset-forged at room temperature to a thickness of 75 mm. Teflon films of 150 x 150 mm2 can thickness of 1.2 mm were used as lubricants for frictionless upsetting (alternatively frictionless upset-forging can also be performed at 300 deg. C). Thereafter cold rolling with a roll diameter of 915 mm was performed in sixteen passes with partial reductions of 12% per pass along four directions under an angle of 45°.

The paragraph beginning at line 14 of page 11 has been amended as follows:

Coupons across the thickness of the rolled billet were cut from central, mid-radius and external areas and annealed at different temperatures during 1 hours (h) and investigated for structure and texture and photomicrographs thereof are shown in FIGS. 1-6. FIGS. 1-3 are photomicrographs of the center, mid-radial and edge, respectively, showing the fine grain structure of a tantalum target. FIGS. 4-6 are graphs showing ~~(100) cubic texture~~ {100} crystallographic orientation at the center, mid-radial and edge--.

The paragraph beginning at line 9 of page 12 has been amended as follows:

Step 1: Anneal the billet in vacuum.

The paragraph beginning at line 11 of page 12 has been amended as follows:

Step 2: Upset-forge billet using teflon as a solid lubricant at room temperature or at 527F to specific height required for rolling.

The paragraph beginning at line 13 of page 12 has been amended as follows:

Step 3: Fly-cut surfaces of the forged billet.

The paragraph beginning at line 16 of page 12 has been amended as follows:

Step 5: Anneal in vacuum to obtain a fine grain size and uniform texture.

The paragraph beginning at line 20 of page 12 has been amended as follows:

Step 1: Upset-forge using teflon to a height such that Mo = 1.0.

The paragraph beginning at line 23 of page 12 has been amended as follows:

Step 3: Upset-forge billet using teflon to a final height as required for rolling operation.

The paragraph beginning at line 25 of page 12 has been amended as follows:

Step 4: Fly-cut the surfaces of the forged billet.

The paragraph beginning at line 32 of page 12 has been amended as follows:

Step 1: Anneal the billet in vacuum.

The paragraph beginning at line 3 of page 13 has been amended as follows:

Step 3: Fly-cut surfaces of the forged billet.

The paragraph beginning at line 6 of page 13 has been amended as follows:

Step 5: in vacuum to obtain a fine grain size and uniform texture.

The paragraph beginning at line 13 of page 13 has been amended as follows:

Step 1: Anneal the billet in vacuum.

The paragraph beginning at line 12 of page 13 has been amended as follows:

Step 2: Upset-forge billet ~~uisng~~ using teflon as a solid lubricant at room temperature or at 572F.

The paragraph beginning at line 13 of page 13 has been amended as follows:

Step 3: Fly-cut surfaces of the forged billet.

In the Claims

The claims have been amended as follows. Underlines indicate insertions and ~~strikeouts~~ indicate deletions.

1-4 (cancelled)

New claims 5-31 (listed above) are added.